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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:

Group Art Unit: 1754

MICHAEL JOHN HOLLITT et al

Examiner: S. Bos

Serial No.: 08/935,108

Attention: Office of
Petitions (Eugenia Jones)

Filed: September 29, 1997

For: UPGRADING TITANIFEROUS MATERIALS

PETITION UNDER 37 CFR 1.53(e)

Honorable Assistant Commissioner for Patents
Washington, DC 20231

Sir:

Applicants hereby petition under the provisions of 37 CFR 1.53(e) to convert the Continued Prosecution Application filed on April 26, 2001 to a Continuation Application under 37 CFR 1.53(b).

The present application was filed originally on February 14, 1995 as Serial No. 08/379,566. A Continuation Application under 37 CFR 1.53(b) was filed on September 29, 1997, Serial No. 08/935,108, and Continued Prosecution Applications were subsequently filed on June 10, 1998, July 6, 1999, October 18, 2000 and April 26, 2001. The Continued Prosecution Application filed on April 26, 2001 was deemed to be improper

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been filed after May 29, 2000, and therefor a Request for Continued Examination should have been filed. The Continued Prosecution Application was treated as a Request for Continued Examination in accordance with the notice in the Federal Register, Vol. 65, No. 159, page 50093 (August 16, 2000), and because the Continued Prosecution Application did not include a submission under 37 CFR 1.114, it was treated as an improper Request for Continued Examination. As the six month period for response to the Final Office Action mailed October 26, 2000 had expired, the application was deemed to be abandoned.

The aforementioned notice in the Federal Register further states:

In the event that an applicant files a request for a CPA of a utility or plant application that was filed on or after May 29, 2000 (to which CPA practice no longer applies), the Office will automatically treat the improper CPA as a request for continued examination of the prior application (identified in the request for CPA) under new § 1.114 (unless the application has issued as a patent). If an applicant files a request for a CPA of an application to which CPA practice no longer applies and does not want the request for a CPA to be treated as a request for continued examination under § 1.114 (e.g., the CPA is a divisional CPA), the applicant may file a petition under § 1.53(e) requesting that the improper CPA be converted to an application under § 1.53(b). The requirements for such a petition under § 1.53(e) are identical to those set forth in section 201.06(b) of the *Manual of Patent Examining Procedure* (7th ed.1998) (Rev. 1, Feb. 2000) (MPEP) for converting an improper file wrapper continuing (FWC) application under former § 1.62 to an application under § 1.53(b). The

Office will not grant such a petition unless it is before the appropriate deciding official before an Office action has been mailed in response to the request for continued examination under § 1.114 (as the improper CPA is being treated). If an Office action has been mailed in response to the request for continued examination under § 1.114, the applicant should simply file an application under § 1.53(b) within the period for reply to such Office action.

The present situation was thus contemplated in the Federal Register notice in that a request for a CPA was filed in an application to which CPA practice no longer applied. Further, Applicants do not wish the CPA filed on April 26, 2001 to be treated as a Request for Continued Examination, which would result in abandonment of the application, but request that the CPA be converted to a Continuation Application under 37 CFR 1.53(b), as noted in the Federal Register.

The requirements for this petition are identical to those set forth in section 201.06(b) of the *Manual of Patent Examining Procedure* (7th Ed. 1998) (Rev. 1, Feb. 2000), and therefor Applicant has attached hereto:

- 1) The \$130.00 Petition Fee;
- 2) A true copy of the complete nonprovisional application, as filed, designated as the prior nonprovisional application; and
- 3) All amendments entered in the prior nonprovisional

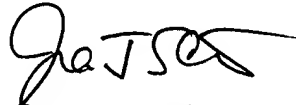
application; and

4) A copy of the executed declaration previously filed.

There are no amendments submitted but not entered in the prior nonprovisional application.

As this petition is submitted according to the guidelines set forth in the Federal Register, favorable action is requested.

Respectfully submitted,



Ira J. Schultz
Registration No. 28666

~~S~~ A

Prior Application: S.N. 08/379,566
Examiner: S. Bos
Art Unit: 1103

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SA
R. Carraway
2-12-98

This is a request for filing a continuation application under 37 CFR 1.160 of pending prior application Serial No. 08/379,566, filed August 9, 1995, of Michael J. Hollitt et al for UPGRADING TITANIFEROUS MATERIALS.

Copies of the papers of the prior application Serial No. 08/381,877 of Michael J. Hollitt et al, as filed, are as follows:

18 pages of specification
3 pages of claims
0 pages of Abstract
0 sheets of drawings
1 page of declaration and power of attorney
filed August 9, 1995

I hereby verify that the attached papers are a true copy of what is shown in my records to be the above-identified prior application, including the declaration originally filed.

Before calculating the filing fee, please enter the attached Preliminary Amendment.

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The filing fee is calculated for this application as follows:

Total Claims	15 - 20 = 0	x \$ 22 = 0	
Independent			
Claims	1 - 3 = 0	x \$ 80 = 0	
Multiple dependent			
Claims.	Yes	x \$260 =	250.00
Basic Filing Fee			770.00
	TOTAL		\$ 770.00

A check in the amount of \$770.00 is enclosed. The Commissioner is hereby authorized to charge payment of any additional filing fees required under 37 CFR 1.16, or any patent application processing fees under 37 CFR 1.17, or credit any overpayment, to Deposit Account 04-753.

Prior application Serial No. 08/379,566 was the designated filing in the United States of International Application PCT/AU/93/00414, filed 12 August 1993.

Amend the specification by inserting before the first line the sentence: --~~This is a continuation of copending Application Serial No. 08/379,566, filed August 9, 1995,~~^{now abandoned.}--

With respect to the prior U.S. Application from which this application claims benefit under 35 USC 120, the inventor in this application is the same and the inventorship for all claims in this application is the same.

The prior application is assigned of record to

TECHNOLOGICAL RESOURCES PTY LIMITED
Melbourne, Victoria 3000 Australia

The power of attorney appears in the original papers of the prior application. A copy of the power filed is enclosed. Address all future communication to:

70110 U.S. PTO
08/935108
09/29/97

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A notification of the filing of this continuation is being filed in the parent application from which this application claims priority under 35 USC § 120.

I hereby declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Ira J. Schultz

Ira J. Schultz
Registration No. 28666
Attorney of Record

Date: 9/29/97

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UPGRADING TITANIFEROUS MATERIALS*ATT BACKGROUND OF THE INVENTION*

ENS A C
This invention relates to the removal of impurities from naturally occurring and synthetic titaniferous materials.

5 The invention is particularly suited to the enhancement of titaniferous materials used in the production of titanium metal and titanium dioxide pigments by means of industrial chlorination systems.

10

Embodiments of the present invention have the common feature of roasting of titaniferous materials in the presence of additives and at temperatures which encourage the formation of a liquid oxide or glassy phase, followed at some stage by
15 cooling and aqueous leaching as steps in an integrated process. Additional steps may be employed as will be described below.

20

In industrial chlorination processes titanium dioxide bearing feedstocks are fed with coke to chlorinators of various designs (fluidised bed, shaft, molten salt), operated to a maximum temperature in the range 700 - 1200C. The most common type of industrial chlorinator is of the fluidised bed
25 design. Gaseous chlorine is passed through the titania and carbon bearing charge, converting titanium dioxide to titanium tetrachloride gas, which is then removed in the exit gas stream and condensed to liquid titanium tetrachloride for further purification and processing.

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The chlorination process as conducted in industrial chlorinators is well suited to the conversion of pure titanium dioxide feedstocks to titanium tetrachloride. However, most other inputs (i.e. impurities in feedstocks) cause difficulties which greatly complicate either the chlorination process itself or the subsequent stages of condensation and purification and disposal of waste. The attached table provides an indication of the types of problems encountered. In addition, each unit of inputs which does not enter products contributes substantially to the generation of wastes for treatment and disposal. Some inputs (e.g. particular metals, radioactives) result in waste classifications which may require specialist disposal in monitored repositories.

Preferred inputs to chlorination are therefore high grade materials, with the mineral rutile (at 95-96% TiO_2) the most suitable of present feeds. Shortages of rutile have led to the development of other feedstocks formed by upgrading naturally occurring ilmenite (at 40-60% TiO_2), such as titaniferous slag (approximately 86% TiO_2) and synthetic rutile (variously 92-95% TiO_2). These upgrading processes have had iron removal as a primary focus, but have extended to removal of magnesium, manganese and alkali earth impurities, as well as some aluminium.

	Elemental Input	Chlorination	Condensation	Purification
5	Fe, Mn	Consumes chlorine, coke, increases gas volumes	Solid/liquid chlorides foul ductwork, make sludges	
10	Alkali & alkali earth metals	Defluidise fluid beds due to liquid chlorides, consume chlorine, coke		
15				
20	Al	Consumes chlorine, coke	Causes corrosion	Causes corrosion, makes sludges
25	Si	Accumulates in chlorinator, reducing campaign life. Consumes coke, chlorine	Can encourage duct blockage. Condenses in part with titanium tetrachloride	May require distillation from product
30				
35	V			Must be removed, by chemical treatment and distillation
40	Th, Ra	Accumulates in chlorinator brickwork, radioactive; causes disposal difficulties		
45				

In the prior art synthetic rutile has been formed from titaniferous minerals, e.g. ilmenite, via various techniques. According to the most commonly applied technique, as variously operated in Western Australia, the titaniferous

mineral is reduced with coal or char in a rotary kiln, at temperatures in excess of 1100 C. In this process the iron content of the mineral is substantially metallised. Sulphur additions are also made to convert manganese impurities partially to sulphides. Following reduction the metallised product is cooled, separated from associated char, and then subjected to aqueous aeration for removal of virtually all contained metallic iron as a separable fine iron oxide. The titaniferous product of separation is treated with 2-5% aqueous sulphuric acid for dissolution of manganese and some residual iron. There is no substantial chemical removal of alkali metals or alkaline earths, aluminium, silicon, vanadium or radionuclides in this process as disclosed or operated. Further, iron and manganese removal is incomplete.

Recent disclosures have provided a process which operates reduction at lower temperatures and provides for hydrochloric acid leaching after the aqueous aeration and iron oxide separation steps. According to these disclosures the process is effective in removing iron, manganese, alkali and alkaline earth impurities, a substantial proportion of aluminium inputs and some vanadium as well as thorium. The process may be operated as a retrofit on existing kiln based installations. However, the process is ineffective in full vanadium removal and has little chemical impact on silicon.

In another prior art invention relatively high degrees of removal of magnesium, manganese, iron and aluminium have been achieved. In one such process ilmenite is first thermally reduced to substantially complete reduction of its ferric oxide content (i.e. without substantial metallisation), normally in a rotary kiln. The cooled, reduced product is then leached under 35 psi pressure at 140-150 C with excess 20% hydrochloric acid for removal of iron, magnesium,

aluminium and manganese. The leach liquors are spray roasted for regeneration of hydrogen chloride, which is recirculated to the leaching step.

5 In other processes the ilmenite undergoes grain refinement by thermal oxidation followed by thermal reduction (either in a fluidised bed or a rotary kiln). The cooled, reduced product is then subjected to atmospheric leaching with excess 20% hydrochloric acid, for removal of the deleterious
10 impurities. Acid regeneration is also performed by spray roasting in this process.

In all of the above mentioned hydrochloric acid leaching based processes impurity removal is similar. Vanadium,
15 aluminium and silicon removal is not fully effective.

In yet another process ilmenite is thermally reduced (without metallisation) with carbon in a rotary kiln, followed by cooling in a non-oxidising atmosphere. The cooled, reduced
20 product is leached under 20 - 30 psi gauge pressure at 130°C with 10 - 60% (typically 18 - 25%) sulphuric acid, in the presence of a seed material which assists hydrolysis of dissolved titania, and consequently assists leaching of impurities. Hydrochloric acid usage in place of sulphuric
25 acid has been claimed for this process. Under such circumstances similar impurity removal to that achieved with other hydrochloric acid based systems is to be expected. Where sulphuric acid is used radioactivity removal will not be complete.

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A commonly adopted method for upgrading of ilmenite to higher grade products is to smelt ilmenite at temperatures in excess of 1500°C with coke addition in an electric furnace, producing a molten titaniferous slag (for casting and

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crushing) and a pig iron product. Of the problem impurities only iron is removed in this manner, and then only incompletely as a result of compositional limitations of the process.

5

In another process titaniferous ore is roasted with alkali metal compounds, followed by leaching with a strong acid other than sulphuric acid (Australian Patent No. AU-B-70976/87). According to this disclosure substantial removal of various impurities is achieved, with "substantial" defined to mean greater than 10%. In the context of the present invention such poor removal of impurities, especially of thorium and uranium, would not represent an effective process. No specific phase structure after roasting is indicated for this process but it is evident from analytical results provided (where product analyses, unlike feed analyses do not sum to 100% and analyses for the alkali metal added are not given) that there may have been significant retention of the additive in the final product. Under the conditions given it is herein disclosed that it is to be expected that alkali ferric titanate compounds which are not amenable to subsequent acid leaching will form. The consequent retention of alkali will render the final product unsuitable as a feedstock for the chloride pigment process.

25

In yet another process a titaniferous ore is treated by alternate leaching with an aqueous solution of alkali metal compound and an aqueous solution of a mineral acid (US Patent No. 5,085,837). The process is specifically limited to ores and concentrates and does not contemplate prior processing aimed at artificially altering phase structures. Consequently the process requires the application of excessive reagent and harsh processing conditions to be even partially effective and is unlikely to be economically

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implemented to produce a feedstock for the chloride pigment process.

5 A wide range of potential feedstocks is available for upgrading to high titania content materials suited to chlorination. Examples of primary titania sources which cannot be satisfactorily upgraded by prior art processes for the purposes of production of a material suited to chlorination include hard rock (non detrital) ilmenites, 10 siliceous leucoxenes, many primary (unweathered) ilmenites and large anatase resources. Many such secondary sources (e.g. titania bearing slags) also exist.

15 Clearly there is a considerable incentive to discover methods for upgrading of titaniferous materials which can economically produce high grade products almost irrespectively of the nature of the impurities in the feed.

SUMMARY OF THE INVENTION

✓ The present invention provides a combination of processing 20 steps which may be incorporated into more general processes for the upgrading of titaniferous materials, rendering such processes applicable to the treatment of a wider range of feeds and producing higher quality products than would otherwise be achievable.

25 Accordingly, the present invention provides a process for upgrading a titaniferous material by removal of impurities which process includes the steps of:-

- 30 (i) heating a titaniferous material to a temperature less than 1300°C to produce a solid titaniferous phase and a liquid oxide or glassy phase in the presence of sufficient of compounds which encourage the formation of the liquid oxide or glassy phase;

5 (ii) cooling the product of step (i) to form a solidified material comprising the titaniferous phase and an impurity bearing phase at a rate sufficient to ensure the susceptibility of the impurity bearing phase to leaching in either an acid or alkaline leachant; and

10 (iii) leaching the solidified material in an acidic or alkaline leachant to leach at least a portion of the impurities.

15 In order to ensure the formation of the solid titaniferous phase and the liquid oxide or glassy phase during the heating step it will normally be necessary to add to the titaniferous material, prior to the heating step, sufficient of a compound that encourages the formation of the liquid oxide or glassy phase. However, in some cases it will not be necessary since the titaniferous material itself may contain sufficient of
20 such a compound.

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DETAILED DESCRIPTION OF THE INVENTION

It has been discovered that the process of the invention can remove iron, magnesium and other alkaline earths, alkalis, manganese, silica, phosphorus, alumina, vanadium, rare
25 earths, thorium and other radioactive elements, which impurities form an almost comprehensive list of impurities in titaniferous mineral sources. From most materials a product purity of greater than 96% TiO₂ can be obtained.

30 Compounds added to the titaniferous material may be mixed therewith by any means ranging from direct mixing of additives prior to charging to thermal treatment to more complex feed preparations such as the formation of agglomerates or nodules of mixed products, to briquette

production from feeds and additives. Many additives will be effective. In particular it is herein disclosed that sodium, potassium, lithium, phosphorus, silicon and boron compounds and minerals (e.g. borax, trona and other alkali metal carbonates, spodumene, caustic soda) will be effective. Additives may be incorporated individually or in combination with other additives.

It is further disclosed herein that the formation of a glassy phase by addition of alkali compounds can be achieved without the formation of alkali titanate phases, reduced alkali titanate phases (e.g. NaTiO_3 - compounds and solid solutions) or alkali ferric titanate phases (e.g. Na(Fe, Al)O_2 - TiO_2 phases known as "bronzes") in roasting. Where such titanate phases form their stability with respect to subsequent leaching steps is such that the final product quality is adversely affected. The incorporation of sufficient quantities of further additives (e.g. boron or phosphorus compounds) which substantially reduce alkali oxide chemical activity can have the effect of eliminating these phases.

Under many circumstances it will be beneficial to incorporate multiple additives into the material to be treated by thermal processing. For example, it is herein disclosed that the simultaneous presence of silica, anhydrous borax and sodium oxide in 1000°C thermally processed material in weight ratios of about 7:1:1 ensures the preferential formation of a glassy phase over other phases containing silica or soda. In this formulation the required borax addition is only just over 10% of the addition which would be required for an equivalent amount of glassy phase where other additives do not act as extenders. Since borax is by far the most expensive additive of the three additives in this case optimum economics are achieved by the use of the extenders.

Thermal processing may be carried out in any suitable device. The production of liquid phases would recommend rotary or grate kilning, but shaft furnaces may also be used and it has
5 been found that fluidised beds can be used under some circumstances. Any gaseous atmosphere conditions may be used, from fully oxidising to strongly reducing. The thermal processing atmosphere should be chosen to most suit other steps in integrated processing. Reducing conditions may be
10 achieved where desired by either the use of a sub stoichiometric firing flame or the addition of coal, char or coke with the thermal processing charge.

Thermal processing residence time at temperature will depend
15 on the nature of the additives and the feed, as well as the operating temperature. Residence times of from 5 minutes to five hours have been effective, allowing thermal processing residence times to be set to most suit other requirements in integrated processing.

20 The level of additive used and the conditions applied in thermal processing should be such that glassy phase formation does not exceed the limitations set by materials handling constraints in the thermal processing step. For example,
25 where glassy phase formation exceeds about 15% by volume of the roasted material it must be anticipated that accretion and bed fusion problems will occur.

Cooling of the thermally treated material should be conducted
30 in such a manner as to limit the reversion of the glassy phase to crystalline phases, i.e. should be at a sufficient rate to a temperature at which the liquid glass solidifies as to ensure the formation of at least a portion of solid glass rather than complete formation of crystalline products.

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Further, cooling should be conducted under an environment appropriate to the conditions of thermal treatment (i.e. reduction processing will require cooling in an oxygen free environment).

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The aqueous leaching step need not necessarily follow directly after the presently disclosed thermal processing step. For example if the thermal processing step is conducted under oxidising conditions it may be optionally followed by a reduction step prior to aqueous leaching. Further, crushing/grinding of the thermally processed material to enhance subsequent leach performance may be undertaken.

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The conditions necessary to conduct effective leaching will depend on the nature of the original feed and the additives. For example, addition of soda ash and borax to siliceous leucoxene in accordance with the present disclosure will result in a product which can be leached in sodium silicate solution formed directly from the thermally treated material; the active leachant in this case is simply water. In other cases up to 100 gpL caustic soda solution or acid will be an effective leachant. Leaching will generally benefit substantially by use of high temperature (e.g. 80°C or above), although it has not been necessary to use pressure leaching to achieve effective conditions. Nevertheless it is presently disclosed that pressure leaching can be effectively and successfully applied. Lower temperatures can also be used, although with penalties in process kinetics.

Leaching may be conducted in any circuit configuration, including batch single or multiple stage leaching, continuous cocurrent multistage leaching, or continuous countercurrent multistage leaching. For most circumstances two stage cocurrent leaching will be most beneficial. Average residence

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time may vary from 30 minutes to 10 hours, depending on process conditions. Any leach vessel capable of providing adequate shear may be used. Simple stirred tank vessels are applicable.

5

At the conclusion of leaching the leach liquor may be separated from the mineral by any suitable means, including thickening, filtration and washing. The mineral product may then pass on to other steps in an integrated process. For example, a further acid leach may follow the disclosed leaching step, particularly where the titaniferous feed has a content of alkalis or alkaline earths.

Other processing steps may be added as necessary or desired. For example, reagent regeneration (e.g. caustic regeneration, hydrochloric acid regeneration, sulphuric acid regeneration) can be used with the process in order to improve process effectiveness or economics. Similarly, a physical separation step may be employed at any stage (e.g. a final magnetic separation to remove grains containing iron, such as chromite).

20

Examples

Example 1

Sodium carbonate addition, corresponding to 4.25% Na_2O by weight, was made to a titania concentrate whose composition is given in Table 1. The mixture was homogenised and pelletised, and the pellets were heated in air to 1000°C for 4 hours. The thus roasted pellets were quenched in liquid nitrogen and then crushed to pass a screen of 200 microns aperture. The crushed roasted pellets were subjected to leaching under reflux with 40 wt% sodium silicate solution ($\text{SiO}_2:\text{Na}_2\text{O}=2,4:1$ by weight) at 4% slurry density. (Sodium silicate solution was used to simulate leaching using water

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as leachant under conditions where the leach liquors are recycled to leaching after solid/liquid separation).

5 Solid/liquid separation was effected by centrifuging, after which the leach residue was washed and calcined at 1000°C for analysis. The analysis of the calcined product is also given in Table 1.

10 The original concentrate was known to contain silica primarily as quartz inclusions in titanate grains. X-ray diffraction analysis after roasting indicated extinction of all crystalline phases containing silica. A glassy phase containing 16% Na₂O, 46% SiO₂, 9% Al₂O₃, 26% TiO₂ and 3% Fe₂O₃ was identified in the roasted material by electron
15 microscopy. Sodium titanates and sodium iron titanium bronze were also identified (along with rutile) by these techniques, indicating that conditions were not optimised.

20 Nevertheless, highly effective concentrate upgrading has been achieved even where the benefits of subsequent acid leaching have not been pursued, illustrating the benefits of the formation of the glassy phase. Substantial removal of silica, alumina and vanadium was achieved.

25 Example 2

This example illustrates the optimisation of additives for both process effectiveness and most economic formulation.

30 In this example titania concentrates of the composition given in Table 2 were used as titaniferous material for treatment. Early work attempting to produce glassy phase with this material by addition of sodium carbonate prior to roasting indicated that glassy phase could easily be produced, but over a wide range of conditions reduced sodium titanate or

sodium iron titanate bronze formation which resulted in sodium retention after leaching could not be easily avoided.

Complete and partial replacement of sodium carbonate by borax was tested.

Two batches of hand pressed pellets were prepared as follows. A 100g sample of the concentrates (previously ground to passing a screen aperture of 30 microns) was blended in each case with 1.1% of the appropriate additive or additive mixture and the resulting blends were pressed into pellets. The first batch was prepared with 1.1 wt% of anhydrous borax addition while the second batch was prepared with addition of 1.1 wt% of 1 : 1 $\text{Na}_2\text{B}_4\text{O}_{10}:\text{Na}_2\text{O}$.

Each batch of pellets was roasted for two hours in a 7 : 1 H_2/CO atmosphere at 1000°C and then removed to cool quickly in the same atmosphere. The roasted pellets were ground to pass a screen aperture of 75 microns for subsequent leaching. Ground roasted pellets were caustic leached under reflux conditions for 6 hours in a 10% NaOH solution at 6.7% solids density. Solid/liquid separation was effected by filtration, and the caustic leached products were washed and dried in preparation for subsequent acid leaching.

The caustic leached residues were acid leached in 15% HCl for 4 hours under reflux, then similarly filtered, washed and dried.

In each case samples of the concentrate and roasted material were submitted for X-ray diffraction analysis. While quartz and various ilmenite, anatase and rutile related phases were identified in the concentrates the only crystalline phases identified in the roasted product were rutile and ilmenite.

All quartz had entered a glassy phase, and no titanate phases which would reduce leach effectiveness were identified.

Analyses of the caustic and acid leach residues in each case, illustrating the effectiveness of the process where optimum conditions are applied, are provided in Table 3.

Example 3

The same pellet formulations as indicated in Example 2 were made up in 350 kg batches in an agglomeration plant and roasted at 30 kg/hr feed rate with 15% brown coal char addition to a final temperature of 1000°C in a small (0.5m diameter) rotary kiln. Residence time above 900°C was approximately 10 minutes. There were no problems with accretions or bed fusion, and after separation from residual char the products had exactly the same properties as the roasted products of Example 2.

Example 4

A commercial titania slag product having the composition indicated in Table 4 was processed as for the processing conditions indicated in Example 2, but with 2 wt% anhydrous borax addition in place of the other additives. The caustic leach was conducted at 165°C under pressure, and a pressure leach with 20% sulphuric acid conducted at 135°C was used in place of the hydrochloric acid leach. The final residue was calcined at 900°C for one hour. The products of this treatment are indicated in Table 4.

Example 5

This example when compared with examples 1 and 2 illustrates the advantages of the formation of a glassy phase.

Concentrates having the composition indicated in Table 1 were

subjected leaching under atmospheric reflux conditions with excess 20% HCl. After separation of the residue from the liquor followed by washing and drying of the residue its composition was as given in Table 5. Clearly there was ineffective removal of virtually all impurities of interest by comparison with the other examples provided herein.

Table 1: Concentrates and Product from Example 1

	wt%	Concentrate	Product
10	TiO ₂	85.8	94.9
	Fe ₂ O ₃	2.25	1.91
	Al ₂ O ₃	1.08	0.63
	SiO ₂	7.62	0.74
	Nb ₂ O ₅	0.30	0.31
15	V ₂ O ₅	0.235	0.02
	Na ₂ O	0.0	1.10

Table 2: Composition of Concentrates
Used in Examples 2 and 3

	wt%
20	TiO ₂ 63.6
	Fe ₂ O ₃ 28.6
	SiO ₂ 3.53
25	Al ₂ O ₃ 0.80
	MgO 0.87
	CaO 0.02
	Cr ₂ O ₃ 0.55
	MnO 1.11
30	V ₂ O ₅ 0.22
	ZrO ₂ 0.26
	P ₂ O ₅ 0.04
	U ₃ O ₈ 0.002
	ThO ₂ 0.01

Table 3: Compositions of Leach Products from Example 2

5	wt%	1.1% Na ₂ B ₄ O ₇ addition		1.1% 1:1 Na ₂ B ₄ O ₇ :Na ₂ O addition	
		Caustic Leach Residue	Acid Leach Residue	Caustic Leach Residue	Acid Leach Residue
10	TiO ₂	66.9	94.3	67.3	94.9
	Fe ₂ O ₃	27.1	30.2	30.6	2.04
	SiO ₂	1.12	0.99	0.55	0.86
	Al ₂ O ₃	0.22	0.17	0.14	0.15
	MgO	0.97	0.08	0.90	0.09
15	CaO	0.05	0.001	0.03	0.001
	Cr ₂ O ₃	0.68	0.69	0.70	0.67
	MnO	1.15	0.06	1.19	0.06
	V ₂ O ₅	0.22	0.15	0.23	0.13
	ZrO ₂	0.27	0.37	0.28	0.38
20	Na ₂ O	0.05	0.02	0.15	0.03
	P ₂ O ₅	0.02	0.02	0.01	0.02
	U ₃ O ₈	0.002	0.002	0.002	0.002
	ThO ₂	0.01	0.003	0.01	0.004

WHAT IS CLAIMED IS
~~CLAIMS~~

1. A process for upgrading a titaniferous material by removal of impurities which process includes the steps of:-

- 5
- (i) heating a titaniferous material to a temperature of less than 1300°C to produce a solid titaniferous phase and a liquid oxide or glassy phase in the presence of sufficient of compounds which encourage the formation of the liquid oxide or glassy phase;
- 10
- (ii) cooling the product of step (i) to form a solidified material comprising the titaniferous phase and an impurity bearing phase at a rate sufficient to ensure the susceptibility of the impurity bearing phase to leaching in either an acid or alkaline leachant; and
- 15
- (iii) leaching the solidified material with an acidic or alkaline leachant to leach at least a portion of the impurities.
- 20

2. A process for upgrading a titaniferous material according to Claim 1 wherein the compounds which encourage the formation of the liquid oxide or glassy phase at a temperature below 1300°C are compounds of sodium, potassium, lithium, phosphorus silicon or boron.

25

3. A process ~~for upgrading a titaniferous mineral~~ according to Claim ¹2, wherein the ~~compound of sodium is~~ ^{additive comprises} caustic soda.

30

4. A process ~~for upgrading a titaniferous mineral~~ according to Claim ¹2, wherein the ~~compound of sodium is~~ ^{additive comprises} sodium carbonate.

5. A process ~~for upgrading a titaniferous mineral~~¹⁸ according to Claim ~~2~~¹⁸, wherein the ~~compounds include~~^{additive comprises} borax.

5 6. A process ~~for upgrading a titaniferous mineral~~¹⁹ according to Claim ~~2~~¹⁹, wherein the ~~compounds comprise~~^{additive comprises} a mixture of soda ash and borax.

Sub C1
10 7. A process according to Claim 6, wherein the titaniferous material is heated to a maximum temperature of 1000°C for a period which avoids substantial reduction to metal of contained iron oxides.

Sub B2
15 8. A process according to Claim 7, wherein the solidified material is leached with water.

20 9. A process according to Claim 7, wherein the solidified material is leached with a recycled solution of leach liquor containing sodium silicate and borax to form a leachate and a residue.

C
C 25 10. A process according to Claim 9, wherein the leachate is separated from the residue and the residue is leached with hydrochloric acid ~~having an acid strength in a range from 1 to 20% hydrochloric acid.~~

Sub C2
30 11. A process according to claim 1 wherein the compounds include compounds which extend the effect of other compounds.

C 12. A process according to claim 11, wherein a compound which extends the effect of other compounds is borax.

C 13. A process according to claim ¹⁷ ~~wherein sufficient compounds are present to avoid the formation of titanate~~^{additive is}

DECLARATION FILED WITH U.S. DESIGNATED OFFICE UNDER 35 U.S.C. 371(c)(4)

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I verily believe I am the original, first and sole inventor (if only one name is listed below) or a joint inventor (if plural inventors are

named below) of the invention entitled: "UPGRADING TITANIFEROUS MATERIALS"

described and claimed in international application no. PCT/AU93/00414 filed 12 August 1993

and as amended on _____ (if any), which I have reviewed and for which I solicit a United States patent;

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

EARLIEST FOREIGN APPLICATION(S), IF ANY, FILED WITHIN 12 MONTHS PRIOR TO THE INTERNATIONAL APPLICATION				
COUNTRY	APPLICATION NUMBER	DATE OF FILING (day, month, year)	DATE OF ISSUE (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. 119
AUSTRALIA	PL 4105	14/12/92		<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
AUSTRALIA	PL 7193	10/2/93		<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
ALL FOREIGN APPLICATIONS, IF ANY, FILED MORE THAN 12 MONTHS PRIOR TO THE INTERNATIONAL APPLICATION				

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.) _____ (Filing Date) _____ (Status—patented, pending, abandoned)

(Application Serial No.) _____ (Filing Date) _____ (Status—patented, pending, abandoned)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (List name and registration number)

Donald L. Dennison Reg. No. 19920

Burton Scheiner Reg. No. 24018

William H. Meserole Reg. No. 20833

Ira J. Schultz Reg. No. 28666

David Pollack Reg. No. 20478

Daniel K. Dorsey Reg. No. 32520

SEND CORRESPONDENCE TO:

DENNISON, MESEROLE, POLLACK & SCHEINER
1745 Jefferson Davis Highway Suite 612
Arlington, Virginia 22202

DIRECT TELEPHONE CALLS TO:

(name and telephone number)

703-412-1155

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor Michael John HOLLITT

Inventor's signature [Signature] Date 7/8/95

Residence 80 Tyne Street, Box Hill North, Victoria 3129 Citizenship Australian

Post Office Address 80 Tyne Street, Box Hill North, Victoria 3129 Australia

Full name of second joint inventor, if any Ross Alexander McCLELLAND

Second Inventor's signature [Signature] Date 7/8/95

Residence Lot 136 Murrawong Road, Maryknoll, Victoria 3812 Australia Citizenship Australian

Post Office Address Lot 136 Murrawong Road, Maryknoll, Victoria 3812 Australia

Full name of third joint inventor John Roger TUFFLEYInventor's signature [Signature]

Date

-8-95Residence 15 Hill Street, Burnside, South Australia Citizenship AustralianPost Office Address 12 Hill Street, Burnside, South Australia 5066 Australia

Full name of fourth joint inventor _____

Second inventor's signature _____

Date _____

Residence _____

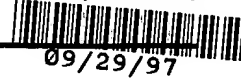
Citizenship _____

Post Office Address _____

20035108-092997

70110 U.S. PTO
08/935108

PATENT APPLICATION SERIAL NO. _____



09/29/97

U.S. DEPARTMENT OF COMMERCE
PATENT AND TRADEMARK OFFICE
FEE RECORD SHEET

D. Cassaway
2-9-98

12/11/1997 BALEXAND 00000005 08935108
01 FC:101 770.00 OP

Dkt. 94150CONT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

MICHAEL J. HOLLITT et al

Continuation Application of:
Serial No. 08/379,566

Filed: concurrently herewith

For: UPGRADING TITANIFEROUS MATERIAL

3/B
D. Garraway
2-12-98

PRELIMINARY AMENDMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Before calculation of the filing fee, please amend
the above application as follows:

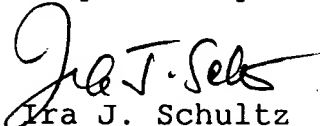
IN THE CLAIMS:

Please cancel Claim 15 without prejudice or
disclaimer of the subject matter thereof.

REMARKS

Claims 1 through 14 are submitted for initial examination.

Respectfully submitted,



Ira J. Schultz
Registration No. 28666

ALL INFORMATION CONTAINED HEREIN IS UNCLASSIFIED

LAW OFFICES
DENNISON, MESEROLE, POLLACK & SCHEINER
612 CRYSTAL SQUARE 4
1745 JEFFERSON DAVIS HIGHWAY
ARLINGTON, VIRGINIA 22202

703 412-1155

#6/C
3/10/98

Dkt. 94150CONT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Group Art Unit: 1103

MICHAEL J. HOLLITT et al

Serial No. 08/935,108

Filed: September 29, 1997

For: UPGRADING TITANIFEROUS MATERIAL

SECOND PRELIMINARY AMENDMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Please amend the above-identified application as follows:

IN THE SPECIFICATION:

Page 1, between lines 1 and 3, insert:

--BACKGROUND OF THE INVENTION--.

Page 7, between lines 17 and 19, insert:

--SUMMARY OF THE INVENTION--.

Page 8, between lines 20 and 22, insert:

--DETAILED DESCRIPTION OF THE INVENTION--.

IN THE CLAIMS:

Page 19, line 1, change "CLAIMS" to

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--WHAT IS CLAIMED IS:--.

Claim 3, line 1, delete "for upgrading a titaniferous mineral";

line 2, change "2" to --18--; change "compound of sodium is" to --additive comprises--.

Claim 4, line 1, delete "for upgrading a titaniferous mineral";

line 2, change "2" to --18--; change "compound of sodium is" to --additive comprises--.

Claim 5, line 1, delete "for upgrading a titaniferous mineral";

line 2, change "2" to --18--; change "compounds include" to --additive comprises--.

Claim 6, line 1, delete "for upgrading a titaniferous mineral";

line 2, change "2" to --18--; change "compounds comprise" to --additive comprises--.

7.(Amended) A process according to Claim 6, wherein the titaniferous material contains an iron oxide impurity and is heated to a maximum temperature of 1000°C for a period which avoids substantial reduction to metal of [contained] the iron [oxides] oxide impurity.

Claim 10, line 2, after "with" insert --1-20 wt.%--;

lines 3-4, delete "having...hydrochloric acid--.

11.(Amended) A process according to claim [1] 17, wherein the [compounds include] additive includes compounds which extend the effect of other compounds in the additive.

Claim 12, line 1, after "11" insert --,--.

Claim 13, line 1, change "1" to --17,--;

line 2, change "compounds are" to --additive is--.

Claim 14, line 1, change "1" to --17,--.

Please cancel Claims 1, 2 and 16 without prejudice or disclaimer of the subject matter thereof, and insert the following new claims:

17. A process for upgrading of a titaniferous material by removal of impurities, comprising the steps of:

i) heating a titaniferous material containing impurities under reducing conditions at a temperature of about 1000°C to less than 1300°C in the presence of an additive which promotes formation of a liquid oxide phase containing said impurities at said temperature, to form a solid titaniferous phase and a liquid oxide phase containing said impurities;

ii) cooling the heated solid titaniferous material and liquid oxide phase to form a solidified material comprising a titaniferous phase and an impurity containing phase that is leachable in an acid or alkaline leachant; and

iii) leaching the solidified material with an acid leachant or an alkaline leachant to leach at least a portion of said impurities.

18. A process according to claim 17, wherein the additive comprises at least one compound selected from the group consisting of compounds of sodium, potassium, lithium, phosphorus, silicon and boron.

19. A process according to claim 17, wherein the solidified material is leached with an alkaline leachant.

20. A process according to claim 19, further comprising leaching the alkaline leached solidified material with an acid leachant.

21. A process according to claim 17, wherein the alkaline leachant is caustic soda.

22. A process according to claim 17, wherein the titaniferous material is heated to a temperature of at least 1000°C.

23. A process according to claim 17, wherein the titaniferous material is heated under reducing conditions.

24. A process according to claim ^{cancelled} 23, wherein the titaniferous material is heated in the presence of a solid ^{New} carbonaceous material. .

25. A process upgrading of a titaniferous material by removal of impurities, comprising the steps of:

03
i) heating a titaniferous material containing impurities under reducing conditions at a temperature of less than 1300°C in the presence of an amount of an additive which promotes formation of a liquid oxide phase containing said impurities at said temperature, to produce thereby a solid titaniferous phase and a liquid oxide phase containing said impurities;

ii) cooling the heated solid titaniferous material and liquid oxide phase to form a solidified material comprising a titaniferous phase and an impurity containing phase that is leachable in an acid or alkaline leachant; and

iii) leaching the solidified material with an acid leachant or an alkaline leachant to leach at least a portion of said impurities.

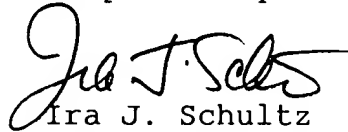
26. A process according to Claim 25, wherein additive is present in an amount of about 1% by weight of the titaniferous material.

3
27. A process according to Claim 25, wherein the additive is present in an amount such that the liquid oxide phase does not exceed about 15% by volume of the titaniferous material.

REMARKS

The above claims are submitted for initial examination.

Respectfully submitted,



Ira J. Schultz
Registration No. 28666

1521
06/18/98

Docket No. PATENT 94150CONT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Michael J. HOLLITT et al

Group: 1103

Serial No.: 08/935,108

Examiner:

Filed: September 29, 1997

For: UPGRADING TITANIFEROUS MATERIAL

AMENDMENT TRANSMITTAL

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Transmitted herewith is an amendment in the above-identified application.

The fee has been calculated as follows:

	Claims Remaining After Amendment		Highest Previously Paid For		Extra		Small Entity	or	Large Entity	
Total	23	-	20	=	3	x	\$11 =		x	\$22 = \$66.00
Indep	2	-	3	=	0	x	\$41 =		x	\$82 =
First presentation of multiple depend claim + \$135 =									+ \$270 =	
TOTAL ADDITIONAL FEE										\$ 66.00

____ Small entity has previously been established and is still applicable.

____ Small entity declaration is enclosed herewith.

____ A Petition for ____ month(s) extension of time is enclosed.

XX A check in the amount of \$66.00 is enclosed to cover the additional claims fee (and/or extension of time fee).

XX The Commissioner is hereby authorized to charge payment of any additional claims fees required under 37 CFR §1.16 or processing fees under 37 CFR §1.17, or credit any overpayment, to Deposit Account 04-0753. A duplicate copy of this sheet is enclosed.

Date 06.00 1998

Ira J. Schultz
Ira J. Schultz, Reg. 28666

(703)412-1155 Ext. 23

GROUP 1103 RECEIVED
98 OCT 17 PM 12:58

LAW OFFICES
DENNIS M. MESEROLE, POLLACK & SCHEINER
1700 JEFFERSON DAVIS HIGHWAY
ARLINGTON, VIRGINIA 22202
703 412-1155
02/18/1998
01 FEB 1998
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103-60000
131-79000

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6/3/98
#101WB
6/17/98

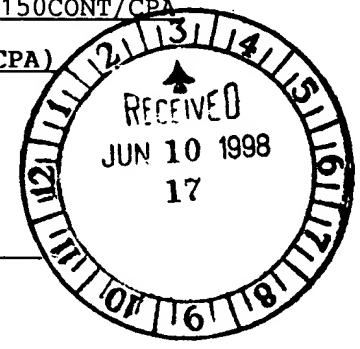
Attorney Dkt.: 94150CONT/CPA

CONTINUED PROSECUTION APPLICATION (CPA)

REQUEST TRANSMITTAL

Honorable Commissioner of Patents and Trademarks
Box CPA
Washington, D.C. 20231

First Named Inventor: Michael J. Hollitt



NOTES

Filing Qualifications: The prior application identified herein must be a nonprovisional application that is either (1) complete as defined by 37 CFR 1.51(b) and filed on or after June 8, 1995, or (2) the national stage of an international application in compliance with 35 U.S.C. 371 and filed on or after June 8, 1995.

CIP Not Permitted: A continuation-in-part cannot be filed as a CPA under 37 CFR 1.53(d), but must be filed under 37 CFR 1.53(b).

Express Abandonment of Prior Application: The filing of this CPA is a request to expressly abandon the prior application as of the filing date of the request for a CPA. 37 CFR 1.53(b) must be used to file a continuation, divisional or continuation-in-part of an application that is not to be abandoned.

Access to Prior Application: The filing of this CPA will be construed to include a waiver of confidentiality by the applicant under 35 U.S.C. 122 to the extent that any member of the public who is entitled under the provisions of 37 CFR 1.14 to access to, copies of, or information concerning, the prior application may be given similar access to, copies of, or similar information concerning, the other application or applications in the file jacket.

35 U.S.C. 120 Statement: In a CPA, no reference to the prior application is needed in the first sentence of the specification and none should be submitted. If a sentence referencing the prior application is submitted, it will not be entered. A request for a CPA is the specific reference required by 35 U.S.C. 120 and to every application assigned the application number identified in such request, 37 CFR 1.78(a).

Sir:

This is a request under 37 CFR 1.53(d) for a continued prosecution application (CPA) of prior Serial Number 08/935,108, filed on September 29, 1997, entitled UPGRADING TITANIFEROUS MATERIALS

This request is for a:

XX Continuation

 Divisional

NEW OFFICES
DENNISON
01-FC:115
02-FC:108
03-FC:111
RESERVED, POLLACK & SCHEINER
772 CRISTAL SQUARE 4
ARLINGTON, VIRGINIA 22202

412-1155

00000010 08935108

110.00 OP
66.00 OP
790.00 OP

Enclosed are:

XX The Preliminary Amendments previously filed on 9/29/97 AND 2/17/98 in the prior nonprovisional application. These Amendments are to be entered before initial examination.

☐ Preliminary Amendment

☐ New Power of Attorney or Authorization of Agent

☐ Information Disclosure Statement

This application is filed by fewer than all the inventors named in the prior application, 37 CFR 1.53(d)(4):

☐ PLEASE DELETE the following inventor(s) named in the prior nonprovisional application:

The filing fee is calculated for this application as follows:

	(Small Entity)	(Large Entity)
BASIC FEE	\$395	\$790
Total Claims	<u>23</u> - 20 = <u>3</u> x \$11 = <u> </u>	x \$ 22 = <u>66</u>
Independent		
Claims	<u>2</u> - 3 = <u>0</u> x \$41 = <u> </u>	x \$ 82 = <u> </u>
Multiple dependent		
Claims.	+ \$135 = <u> </u>	+ \$270 = <u> </u>
TOTAL	\$ <u> </u>	\$856.00

Small Entity Status:

☐ A small entity statement is enclosed.

☐ A small entity statement was filed in the prior application and such status is still proper and desired.

☐ Is no longer claimed.

An extension of time for one month in the prior application is enclosed together with the required fee of \$110.00.

A check in the amount of \$ 966.00 is enclosed to cover the filing fee and the extension of time fee. The Commissioner is hereby authorized to charge payment of any additional filing fees required under 37 CFR 1.16, 37 CFR 1.17, or 37 CFR 1.18, or credit any overpayment, to Deposit Account 04-0753.

6/10/98
Date.

Ira J. Schultz
Ira J. Schultz
Registration No. 28666
Attorney of Record

DENNISON, MESEROLE, POLLACK & SCHEINER
Crystal Square 4, Suite 612
1745 Jefferson Davis Highway
Arlington, VA 22202
(703) 412-1155 Ext. 23
(703) 412-1161 (fax)

Dkt. 94150CONT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

MICHAEL JOHN HOLLITT, et al

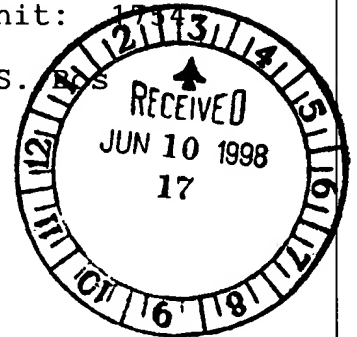
Group Art Unit: 17343

Serial No.: 08/935,108

Examiner: S. P. S.

Filed: September 27, 1997

For: UPGRADING TITANIFEROUS MATERIALS



PETITION UNDER 37 CFR § 1.136

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Petition is herein made under the provisions of 37 CFR 1.136 for an extension of time for one month for response to the Office action of February 13, 1998. The appropriate fee set forth in 37 CFR 1.17 is filed herewith.

Please charge any additional fees or credit overpayment to Deposit Account No. 04-0753. A duplicate copy of this paper is enclosed.

Respectfully submitted,

A handwritten signature in cursive script, appearing to read "Ira J. Schultz".

Ira J. Schultz
Registration No. 28666

Dkt. 94150CONT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

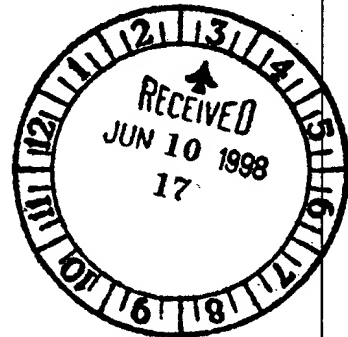
Group Art Unit: 1103

MICHAEL J. HOLLITT et al

Serial No. 08/935,108

Filed: September 29, 1997

For: UPGRADING TITANIFEROUS MATERIAL



SECOND PRELIMINARY AMENDMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Please amend the above-identified application as follows:

IN THE SPECIFICATION:

Page 1, between lines 1 and 3, insert:

--BACKGROUND OF THE INVENTION--.

Page 7, between lines 17 and 19, insert:

--SUMMARY OF THE INVENTION--.

Page 8, between lines 20 and 22, insert:

--DETAILED DESCRIPTION OF THE INVENTION--.

IN THE CLAIMS:

Page 19, line 1, change "CLAIMS" to

DUPLICATE

--WHAT IS CLAIMED IS:--.

Claim 3, line 1, delete "for upgrading a titaniferous mineral";

line 2, change "2" to --18--; change "compound of sodium is" to --additive comprises--.

Claim 4, line 1, delete "for upgrading a titaniferous mineral";

line 2, change "2" to --18--; change "compound of sodium is" to --additive comprises--.

Claim 5, line 1, delete "for upgrading a titaniferous mineral";

line 2, change "2" to --18--; change "compounds include" to --additive comprises--.

Claim 6, line 1, delete "for upgrading a titaniferous mineral";

line 2, change "2" to --18--; change "compounds comprise" to --additive comprises--.

7.(Amended) A process according to Claim 6, wherein the titaniferous material contains an iron oxide impurity and is heated to a maximum temperature of 1000°C for a period which avoids substantial reduction to metal of [contained] the iron [oxides] oxide impurity.

Claim 10, line 2, after "with" insert --1-20 wt.-%--;

lines 3-4, delete "having...hydrochloric acid--.

11.(Amended) A process according to claim [1] 17, wherein the [compounds include] additive includes compounds which extend the effect of other compounds in the additive.

Claim 12, line 1, after "11" insert --,--.

Claim 13, line 1, change "1" to --17,--;

line 2, change "compounds are" to --additive is--.

Claim 14, line 1, change "1" to --17,--.

Please cancel Claims 1, 2 and 16 without prejudice or disclaimer of the subject matter thereof, and insert the following new claims:

--17. A process for upgrading of a titaniferous material by removal of impurities, comprising the steps of:

i) heating a titaniferous material containing impurities under reducing conditions at a temperature of about 1000°C to less than 1300°C in the presence of an additive which promotes formation of a liquid oxide phase containing said impurities at said temperature, to form a solid titaniferous phase and a liquid oxide phase containing said impurities;

ii) cooling the heated solid titaniferous material and liquid oxide phase to form a solidified material comprising a titaniferous phase and an impurity containing phase that is leachable in an acid or alkaline leachant; and

iii) leaching the solidified material with an acid leachant or an alkaline leachant to leach at least a portion of said impurities.

18. A process according to claim 17, wherein the additive comprises at least one compound selected from the group consisting of compounds of sodium, potassium, lithium, phosphorus, silicon and boron.

19. A process according to claim 17, wherein the solidified material is leached with an alkaline leachant.

20. A process according to claim 19, further comprising leaching the alkaline leached solidified material with an acid leachant.

21. A process according to claim 17, wherein the alkaline leachant is caustic soda.

22. A process according to claim 17, wherein the titaniferous material is heated to a temperature of at least 1000°C.

23. A process according to claim 17, wherein the titaniferous material is heated under reducing conditions.

24. A process according to claim 23, wherein the titaniferous material is heated in the presence of a solid carbonaceous material.

25. A process upgrading of a titaniferous material by removal of impurities, comprising the steps of:

i) heating a titaniferous material containing impurities under reducing conditions at a temperature of less than 1300°C in the presence of an amount of an additive which promotes formation of a liquid oxide phase containing said impurities at said temperature, to produce thereby a solid titaniferous phase and a liquid oxide phase containing said impurities;

ii) cooling the heated solid titaniferous material and liquid oxide phase to form a solidified material comprising a titaniferous phase and an impurity containing phase that is leachable in an acid or alkaline leachant; and

iii) leaching the solidified material with an acid leachant or an alkaline leachant to leach at least a portion of said impurities.

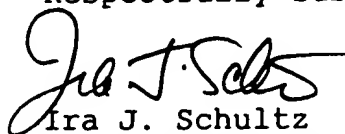
26. A process according to Claim 25, wherein additive is present in an amount of about 1% by weight of the titaniferous material.

27. A process according to Claim 25, wherein the additive is present in an amount such that the liquid oxide phase does not exceed about 15% by volume of the titaniferous material.--

REMARKS

The above claims are submitted for initial examination.

Respectfully submitted,



Ira J. Schultz
Registration No. 28666

Dkt. 94150CONT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

MICHAEL JOHN HOLLITT et al

Group Art Unit: 1754

Serial No.: 08/935,108

Examiner: S. Bos

Filed: September 29, 1997

For: UPGRADING TITANIFEROUS MATERIALS

15/D
W.M.
12/4/98

AMENDMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

In response to the Office action mailed June 24,
1998, please amend the above-identified application as
follows:

IN THE ABSTRACT:

Please add the following Abstract of the Disclosure:

--ABSTRACT OF THE DISCLOSURE

DI

A process for upgrading titaniferous material by removal of impurities contained in the material, especially radionuclides. The process involves heating the titaniferous material to a temperature of about 1000°C to less than 1300°C under reducing conditions, to form a solid titaniferous phase and an impurity - containing liquid oxide phase in the presence of a material that promotes the formation of the liquid oxide phase. The solid titaniferous phase and liquid oxide phase are cooled at a rate that maintains the separate impurity - containing phase in an amorphous state, which can be leached within acid or alkali to remove the impurities. Materials that promote the formation of the desired liquid oxide phase include compounds of alkali metals and boron, for example borax, caustic soda, soda ash and silica.--

IN THE CLAIMS:

D2
SUB E1
8. (Amended) a process according to Claim 7, wherein the solidified material is a leached with water containing dissolved product of said heating step i).

Please cancel Claims 22 and 23 without prejudice or disclaimer of the subject matter thereof.

REMARKS

The Office Action of June 24, 1998 has been carefully considered.

Objection has been raised that this application does not contain an Abstract. And Abstract has now been added.

Claims 8, 22 and 23 have been objected to under 37 CFR 1.75(c), as being of improper dependent form.

The recitation of claim 8 is supported by the specification at p. 11, lines 14 - 20. The specification recites that the conditions for conducting effective leaching will depend upon the nature of the original feed and the additives, and specifically states that where, for example, the additives are soda ash and borax, the product can be leached in a sodium silicate solution formed directly from the thermally treated material. This is clearly an *in situ*

formation of alkaline material which serves as a leachant when water is added. In accordance with this disclosure of the specification, claim 8 has now been amended to specifically recite that the leachant is water containing the product of the heating step. Since the product of the heating step disclosed on page 11 is an alkali (although an acid is not excluded), claim 8 properly limits the subject matter of and depends from claim 17.

Claims 22 and 23 have now been canceled.

Claims 3-14 and 17-27 have been rejected under 35 USC § 103(a) over Heikel '916 or France '777 or Leary '438 or Pollard '934 or Stewart '099 or Stewart '929, each in view of Chao '837. Applicants submit that the presently claimed invention is patentable over the cited combination of references.

Claim 17 has been amended in the manner discussed at the interview of September 15, 1997, to recite that the heating step takes place under reducing conditions and at a temperature of about 1000°C to less than 1300°C. The claim further recites that the heating step takes place in the presence of an additive which promotes formation of a liquid oxide phase containing the impurities and concurrently, a

solid titaniferous phase. The heated material is cooled, the liquid oxide phase forming a solidified material comprising a titaniferous phase and an impurity containing phase that is leachable in an acid or alkaline leachant. Solidified material is then leached with an acid leachant or an alkaline leachant to leach at least a portion of said impurities.

The amendments to claim 17 were not entered in the parent application because they were made after Final Rejection and raised new issues for further consideration and search. While the amendments have now been entered in this continuation application, the present Office Action neither reflects the results of the interview as indicated on the Interview Summary, nor provides any reason why the Examiner now disagrees with the specific points raised at the interview.

Thus, the Heikel reference discloses a process for preparing pigmentary titanium dioxide in which a roasting step is carried out under oxidizing conditions, as disclosed for example, at column 5, lines 22-23. At column 3, lines 42-44, Heikel discloses that during the roasting step in the preferred embodiment the titanium dioxide reacts with sodium hydroxide to form sodium titanate. Sodium titanate is

extremely difficult to leach and the undesirability of such titanates is disclosed in the paragraph beginning at line 9 on page 9 of the present application. The difficulty of leaching titanates is reflected by the extreme digestion conditions used by Heikel.

Heikel clearly differs from the claimed invention in the use of oxidizing conditions in the roasting step; in the claimed invention, the heating step takes place under reducing conditions. The Interview Summary states that the proposed amendment appeared to overcome Heikel and no reason is presented why a reference which teaches heating under oxidizing conditions should render obvious a process step of heating under reducing conditions.

France '777 discloses a process in which a titaniferous material is heated at a temperature in the range of 1300 to 1700°C under conditions which form a molten slag containing titanium dioxide and other oxides and molten iron. The molten slag and molten metal are then separated and allowed to cool, and the molten slag is leached in acid.

The French patent discloses heating a titaniferous material at a temperature greater than 1300°C, whereas the presently claimed invention uses temperatures of less than

1300°C. Moreover, the presently claimed invention forms a solid titaniferous phase, whereas there is no solid titaniferous phase retained under the heating conditions of the French patent. Indeed, the objective of the French patent is to not retain a solid titaniferous phase and the patent clearly discloses that the slag, i.e. the titaniferous phase is molten; see the paragraph bridging pages 5 and 6.

Since the claimed invention clearly recites a solid titaniferous phase, obtained at a temperature of less than 1300°C, while the reference clearly teaches a liquid titaniferous phase obtained at a temperature of above 1300°C, Applicants submit that the claimed invention is clearly distinguished from the France '777 reference.

The Leary patent discloses a process in which titanium and iron compounds are mixed with a flux and heated to a temperature within the range of 350 to 650°C. Under these conditions, the titanium values dissolve in the flux, which is molten, while the iron forms a solid alkali metal ferrate compound dispersed in the flux. There is no disclosure or suggestion in the Leary patent that the titaniferous material should be heated in the presence of an

additive which promotes the formation of a liquid oxide phase containing impurities and a solid titaniferous phase.

The amendment of claim 17 to recite a temperature range of about 1000°C to less than 1300°C clearly distinguishes over the process of Leary. Moreover, it is clear that carrying out the process of Leary would not fall within the presently claimed invention, since the titaniferous material would be present in the liquid flux phase rather than in a solid phase dispersed in a liquid oxide phase.

Applicants note that the presently claimed distribution of titaniferous and impurity materials may be a result of the amounts of titaniferous material and liquid oxide material which are mixed together. Accordingly, Claim 25 recites that the heating step takes place in the presence of an amount of an additive which promotes formation of a liquid oxide phase containing the impurities at the temperature of heating, to produce thereby a solid titaniferous phase and a liquid oxide phase containing the impurities. Claim 26 recites that the additive is present in an amount of about 1% by weight of the titaniferous material, as disclosed in Example 2 of the specification (1.1% by weight) and Claim 27 recites that the additive is present in

an amount such that the liquid oxide phase does not exceed about 15% by volume of the titaniferous material, as disclosed in the present specification at page 10, lines 21-27.

The Pollard et al reference and the Stewart et al references all disclose heating a titaniferous material in the presence of a flux which is a chloride. According to these references, alkali metal chlorides and alkaline earth metal chlorides are used. The materials used by Pollard and Stewart et al are not additives which promote the formation of a liquid oxide phase according to the presently claimed invention; those materials, as can be seen from the present examples, are oxides, hydroxides, borates and silicates, otherwise thought of as "glass formers." Thus, the Pollard et al and Stewart et al references do not disclose heating a titaniferous material with an additive which promotes the formation of a liquid oxide phase. This point was noted at the interview, and it is reflected in the Interview Summary that "[R]ecitation of liquid oxide phase appears to exclude the use of chlorides taught by Stewart refs, and Pollard." No reason has been presented in the Office Action why it is now believed that the use of chlorides will promote the formation of a liquid oxide phase.

The Chao et al reference discloses subjecting a TiO_2 - containing ore to two or more leaching treatments. While sequential leaching treatments may be known, Chao et al does nothing to cure the defects of the primary references discussed above.

Accordingly, the references taken individually or in combination do not disclose or suggest heating a titaniferous material under reducing conditions in the presence of an additive which promotes the formation of a liquid oxide phase containing the impurities present in the titaniferous material, forming thereby a solid titaniferous phase and a liquid oxide phase containing impurities, cooling the solid material and liquid phase to form a solidified material, and leaching the solidified material to leach at least a portion of the impurities. To the contrary, Heikel teaches heating under oxidizing conditions rather than reducing conditions, France '777 teaches heating at a higher temperature to obtain a liquid titaniferous phase, Leary teaches heating at a lower temperature to obtain titanium values dissolved in a molten flux, and Stewart et al and Pollard et al teach the use of chloride additives which do not promote the formation of a liquid oxide phase. As shown hereinabove, the claimed

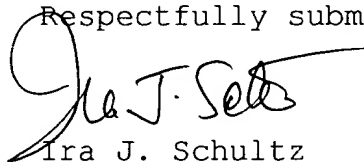
invention is clearly distinguished from each of the references.

Accordingly, withdrawal of this rejection is requested.

In view of the foregoing amendments and remarks, Applicants submit that the present application is now in condition for allowance. An early allowance of the application with amended claims is earnestly solicited.

Finally, Applicants submit herewith a copy of US Patent No. 5,578,109, listed on attached form PTO-1449, a petition for its consideration and the required fee.

Respectfully submitted,



Ira J. Schultz
Registration No. 28666

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Dkt. 94150CONT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

MICHAEL JOHN HOLLITT et al

Group Art Unit: 1754

Serial No.: 08/935,108

Examiner: S. Bos

Filed: September 29, 1997

For: UPGRADING TITANIFEROUS MATERIALS

AMENDMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

In response to the Office action mailed September 27, 1999, please amend the above-identified application as follows:

IN THE CLAIMS:

8. (Twice Amended) ^A process according to Claim 7, wherein the solidified material is ~~leached~~ leached with [water containing dissolved] an aqueous solution of a product of said

EL
heating step i) contained in said solidified material.

Please add the following new claim:

Ed
-28. A process according to claim 8, wherein the product is sodium silicate.

REMARKS

The Office Action of September 27, 1999 has been carefully considered.

Claim 8 continues to be rejected under 35 USC 112, first paragraph, on the basis that claim 8 embodies new matter. It is alleged that the supporting material in the specification at page 11, lines 14-20 does not mention water containing dissolved alkali being used to leach the solidified material.

Applicants concede that the supporting material appears somewhat contradictory, in its statement that the "active leachant... is simply water." However, this paragraph also states that the "addition of soda ash and borax to siliceous leucoxene in accordance with the present disclosure will result in a product which can be leached in sodium silicate solution formed directly from the thermally treated

material..."(emphasis added). Thus, the specification clearly states that 1) the heating step (i) produces a product containing sodium silicate, and 2) the thermally treated material can be leached in a solution of this sodium silicate. Since the next sentence states that the active leachant in this case is simply water, it should be clear to one of ordinary skill in the art that the leachant is obtained by adding water to the sodium silicate produced by the heating step.

In any event, claim 8 has been amended to clarify the steps, reciting that the leaching takes place in an aqueous solution of a product of the heating step (i) contained in the solidified material, which is clearly supported by the specification on page 11 where leaching takes place with an aqueous solution of sodium silicate which is a product of the heating step contained in the solidified material. In addition, new claim 28 specifies that the product is sodium silicate.

Withdrawal of this rejection is requested.

Claim 8 has also been rejected under 35 USC 112, second paragraph, on the basis that there is no antecedent for

the product of step (i). In view of the amendment to claim 8, withdrawal of this rejection is requested.

Claims 3-14, 17-21 and 24-27 have been rejected under 35 USC § 103(a) over France '777 or Stewart '099 or Stewart '929, each in view of Chao '837. Applicants submit that the presently claimed invention is patentable over the cited combination of references.

It is the position of the Office Action that the heating steps disclosed in the cited references "would appear to provide the instantly claimed solid titaniferous phase and the liquid oxide phase since the reactants and temperatures are the same as those instantly claimed."

Claim 17 recites a process including an initial heating step which 1) takes place under reducing conditions and at a temperature of about 1000°C to less than 1300°C, and 2) takes place in the presence of an additive which promotes formation of a liquid oxide phase containing the impurities and concurrently, a solid titaniferous phase. The heated material is cooled, the liquid oxide phase forming a solidified material comprising a titaniferous phase and an impurity containing phase that is leachable in an acid or

alkaline leachant. The solidified material is then leached with an acid leachant or an alkaline leachant to leach at least a portion of these impurities.

France '777 discloses a process in which:

a titaniferous material is heated at a temperature in the range of 1300 to 1700°C under conditions which form a molten slag containing titanium dioxide and other oxides, and molten iron,

the molten slag is separated from the molten iron, and

the slag is leached with sulfuric acid.

The French patent thus discloses heating a titaniferous material at a temperature greater than 1300°C, whereas the presently claimed invention uses temperatures of no more than 1300°C.

Moreover, the heating step of France '777 produces two molten phases, a slag phase and an iron phase. There are numerous references in France '777 which confirm that the heating step produces two molten phases, among them (with reference to the English translation):

Page 4, lines 24-25: "molten mass of metallic iron and a titanium slag";

Page 5, line 20: "a fluid slag";

Page 6, line 1: "a freely-flowing fluid slag";

Page 7, third to last line: "The slag produced is fluid and flows readily."

The heating step of France '777 does not produce a titaniferous phase which is present as a solid phase under the heating conditions, and is therefore fundamentally different from the claimed invention. Indeed, the objective of the French patent is to not retain a solid titaniferous phase.

Since the claimed invention clearly recites a solid titaniferous phase, obtained at a temperature of no more than 1300°C, and the reference clearly teaches a liquid titaniferous phase obtained at a temperature of above 1300°C, Applicants submit that the claimed invention is clearly distinguished from the France '777 reference.

The Stewart et al references disclose heating a titaniferous material in the presence of a flux which is a chloride, typically an alkali metal chloride. The process described by Stewart et al is a segregation-based process

which involves the removal of iron from titaniferous material (ilmenite) with the aid of the molten flux.

In this process, ilmenite is mixed with the flux and a solid carbonaceous reactant (in the presence of hydrogen chloride according to Stewart et al '929). The ilmenite-reactant-flux mixture is placed in a carbon crucible, and is heated to a temperature between 900 and 1200°C. Upon cooling, Stewart et al does not leach, but instead subjects the solid product to a washing operation with a magnetic separation treatment to produce three fractions:

1) a highly magnetic fraction containing over 80% by weight of iron, of sufficient purity for use in steel-making operations;

2) a slightly magnetic fraction containing 10-20% by weight metallic iron mixed with titania, and which could be used as a feedstock for a sulfate pigment process and which could be further upgraded; and

3) a substantially non-magnetic, high purity titanium dioxide which preferably contains less than 3% by weight iron, and which can be used in the chloride pigment process.

The flux used by Stewart et al performs the role of a diffusion medium for the reduction of iron. A relatively large amount of flux is required as it is necessary for the flux to fill at least a large part of the voids within and between grains, and to form bridges between the titaniferous material and the reductant. The flux thereby provides a medium for the reduction and diffusion of reduced iron species for the coalescence of metallic iron into large, physically separable phases.

Thus, Stewart et al does not disclose the formation of a liquid oxide phase containing impurities, and a titaniferous phase which remains solid at the temperature of heating. Moreover, the presently claimed leaching step is considerably different from the washing and magnetic separation step of Stewart et al. In light of these differences, the presently claimed parameters are not mere optimization of prior art processes, as has been alleged, but relate instead to an entirely different process which is not disclosed or suggested by the cited art.

Chao et al has been cited to show the use of acid and alkali leaching treatments in the treatment of TiO_2 ore.

Applicants concede that the use of such leaching is known, but argue strongly that there is no disclosure or suggestion to combine such a treatment with the process of Stewart et al which utilizes a physical separation of iron impurities from titanium dioxide, as is evidenced by the claims of both Stewart et al patents.

As to the France '777 reference, this patent disclosed the use of sulfuric acid extraction on a slag obtained at high temperatures, and there is no suggestion of using an acid extraction step with a process such as Stewart et al which requires a physical separation.

Accordingly, the references taken individually or in combination do not disclose or suggest heating a titaniferous material under reducing conditions at 1000 to 1300°C in the presence of an additive which promotes the formation of a liquid oxide phase containing the impurities present in the titaniferous material, forming thereby a solid titaniferous phase and a liquid oxide phase containing impurities, cooling the solid material and liquid phase to form a solidified material, and leaching the solidified material to leach at least a portion of the impurities. To the contrary, France

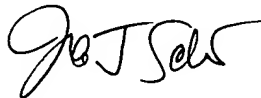
'777 teaches heating at a higher temperature to obtain a liquid titaniferous phase, and Stewart et al teach the use of chloride additives which do not promote the formation of a liquid oxide phase, and utilizes physical separation methods.

Thus, the claimed invention is clearly distinguished from each of the references and withdrawal of this rejection is requested.

In view of the foregoing amendments and remarks, Applicants submit that the present application is now in condition for allowance. An early allowance of the application with amended claims is earnestly solicited.

Finally, Applicants submit herewith a copy of Australian Patent Specification 46,918/72, listed on attached form PTO-1449, a petition for its consideration and the required fee.

Respectfully submitted,



Ira J. Schultz
Registration No. 28666

Docket No. PATENT
94150CONT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Michael John HOLLITT et al

Group: 1754

Serial No.: 08/935,108

Examiner: S. bos

Filed: September 29, 1997

For: UPGRADING TITANIFEROUS MATERIALS

AMENDMENT TRANSMITTAL

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Transmitted herewith is an amendment in the above-identified application.

The fee has been calculated as follows:

	Claims Remaining After Amendment		Highest Previously Paid For		Extra		Small Entity	or	Large Entity	
Total	22	-	21	=	1	x	\$ 9 =	.	x	\$18 = \$18
Indep	3	-	0	=		x	\$39 =		x	\$78 =
First presentation of multiple depend claim + \$130 =									+ \$260 =	
TOTAL ADDITIONAL FEE									\$	\$18

____ Small entity has previously been established and is still applicable.

____ Small entity declaration is enclosed herewith.

XXX A Petition for 3 month(s) extension of time is enclosed.

XXX A check in the amount of \$1,128.00 is enclosed to cover the additional claims fee (and/or extension of time fee), Petition for late IDS fee.

XXX The Commissioner is hereby authorized to charge payment of any additional claims fees required under 37 CFR \$1.16 or processing fees under 37 CFR \$1.17, or credit any overpayment, to Deposit Account 04-0753. A duplicate copy of this sheet is enclosed.

Date

3/24/2000

Ira J. Schultz
Ira J. Schultz, Reg. 28666

FORM PTO-892 (REV. 2-92)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		SERIAL NO. 935108	GROUP/ART UNIT 1103	ATTACHMENT TO PAPER NUMBER 4
NOTICE OF REFERENCES CITED				APPLICANT(S) Hollitt et al		

U.S. PATENT DOCUMENTS							
•	A	DOCUMENT NO.	DATE	NAME	CLASS	SUB-CLASS	FILING DATE IF APPROPRIATE
*	A	4759916	7/88	Heikel	423	71	
*	B	5378438	1/95	Leary	423	80	11-30-92
*	C	4047934	9/77	Pollard et al	75	435	
*	D	3854929	12/74	Stewart et al	423	83	
*	E	3816099	6/74	Stewart et al	423	80	
	F						
	G						
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	I						
	J						
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FOREIGN PATENT DOCUMENTS									
•	L	DOCUMENT NO.	DATE	COUNTRY	NAME	CLASS	SUB-CLASS	PERTINENT SHTS. DWG.	PP. SPEC.
*	L	1066777	6/54	France	—	—	—		
	M								
	N								
	O								
	P								
	Q								

OTHER REFERENCES (Including Author, Title, Date, Pertinent Pages, Etc.)	
R	
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EXAMINER <i>[Signature]</i> Bar	DATE 2-12-98
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* A copy of this reference is not being furnished with this office action.
(See Manual of Patent Examining Procedure, section 707.05 (a).)

#22

Sheet 1 of 1

Form PTO-1449				ATTY DOCKET NO. 94150CONT				SERIAL NO. 08/935,108							
INFORMATION DISCLOSURE CITATION IN AN APPLICATION								APPLICANT <div style="text-align: center;">Michael John HOLLITT et al</div>							
(Use several sheets if necessary)								FILING DATE <div style="text-align: center;">September 29, 1997</div>				GROUP <div style="text-align: center;">1754</div>			

U.S. PATENT DOCUMENTS													
EXAMINER INITIAL	DOCUMENT NUMBER							DATE	NAME	CLASS	SUB CLASS	FILING DATE IF APPROPRIATE	

FOREIGN PATENT DOCUMENTS													
EXAMINER INITIAL	DOCUMENT NUMBER							DATE	COUNTRY	CLASS	SUB CLASS	TRANSLATION	
												YES	NO
SJB	4	6	9	1	8	7	2	3/74	Australia				

OTHER DOCUMENTS (Including Author, Title, Date, Pertinent Pages, etc.)													

EXAMINER <div style="text-align: center;">SJB</div>	DATE CONSIDERED <div style="text-align: center;">4-14-08</div>
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EXAMINER: Initial if citation considered, whether or not citation is in conformance with M.P.E.P. 609; Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.